

PROJECT TITLE : ANALYTICAL INVESTIGATIONS  
PERIOD COVERED : JANUARY 28th - FEBRUARY 22nd  
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GC/MS RESEARCH

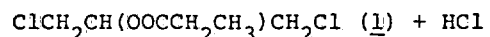
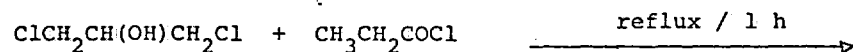
Triacetin ESTROBOND B

Triacetin containing the same amount of glycerol-propionate-diacetate isomer mixture as ESTROBOND B (1) was synthesized by treating glycerol with a mixture of acetic and propionic anhydride (molar ratio 100:99:1) at 139° for 1 hour.

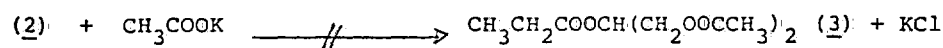
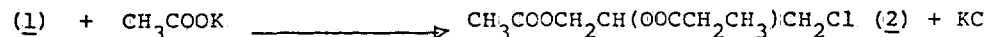
The product will be forwarded to QC for testing as filter additive.

Glycerol-propionate-diacetate isomer synthesis

Glycerol-2-propionate-1,3-diacetate (3) :

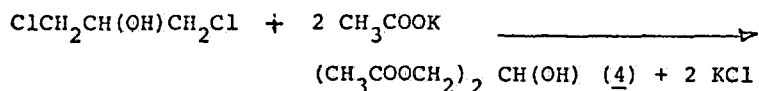


(1) :  $\text{C}_6\text{H}_{10}\text{Cl}_2\text{O}_2$  (185); MS : 135 (M -  $\text{CH}_2\text{Cl}$ , 17), 75 ( $\text{CH}_3\text{CH}_2\text{COOH}_2$ , 53), 57 ( $\text{CH}_3\text{CH}_2\text{CO}$ , 100), 49 ( $\text{CH}_2\text{Cl}^{35}$ , 13), 29 ( $\text{CH}_3\text{CH}_2$ , 24); yield 95 % (GC).



The reaction of (1) with 2 mole of potassium acetate in refluxing ethanol/24 h gave (2) in ~5 % yield; (3) was not formed.

(2) :  $\text{C}_8\text{H}_{13}\text{ClO}_4$  (208); MS : 173 (M -  $\text{Cl}^{35}$ , 1), 159 (M -  $\text{CH}_2\text{Cl}$ , 3), 57 ( $\text{CH}_3\text{CH}_2\text{CO}^+$ , 100), 43 ( $\text{CH}_3\text{CO}$ , 54), 29 ( $\text{CH}_3\text{CH}_2$ , 18).



Treatment of 1,3-dichloro-2-propanol with 2 moles of potassium acetate in refluxing ethanol yielded 80 % (GC) (4).

(4) :  $\text{C}_7\text{H}_{12}\text{O}_5$  (176); MS: 103 (M-  $\text{CH}_3\text{COOCH}_2$ , 33), 74 ( $\text{CH}_3\text{CO}(\text{OH})\text{CH}_2$ , 74), 43 ( $\text{CH}_3\text{CO}$ , 100).

The reaction of (4) with propionic anhydride to (3) is in progress.

#### AMINO ACID ANALYSIS

##### Efficiency of protein hydrolysis

Six probes of pure lysozyme protein of known amino acid profile (2) were hydrolyzed under conditions applied for yeast hydrolysis (6N HCl, 110°C, 48 h) and the individual amino acids analyzed. The number of amino acids per mole lysozyme found, %-recovery and standard deviation s are summarized in Table 1. As can be seen most of the values obtained for the composition of lysozyme are in good agreement with those reported (2).

#### REFERENCES

- (1) E. Lecoultre, PME Research Laboratory, Monthly Progress Report, January 1980.
- (2) L.B. James, J. Chromatogr. 68 (1972) 123.

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Table 1. Amino acid composition of Lysozyme; efficiency of protein hydrolysis

Amino acid	Lysozyme		%	s <sup>b)</sup>
	No. of amino acids per mole of protein (2)	No. of amino acids per mole of protein found <sup>a)</sup>		
Try	6	--	--	--
Lys	6	5.8	97.3	8.0
His	1	1.4	137.2(!)	10.1
Arg	11	12.7	115.1	3.7
Asp	21	22.6	107.6	5.7
Thr	7	6.9	98.2	4.3
Ser	10	9.5	94.9	5.8
Glu	5	5.3	106.4	4.1
Pro	2	3.4	170.0(!)	9.6
Gly	12	13.1	109.3	4.3
Ala	12	12.3	102.9	5.5
½Cys	8	2.9	36.7(!)	1.9
Val	6	6.8	113.3	5.6
Met	2	2.2	109.9	3.2
Ile	6	5.73	95.6	3.7
Leu	8	8.5	106.9	0.8
Tyr	3	3.2	107.9	5.0
Phe	3	3.1	101.9	4.6

a) Mean value of six protein analyses

b) 18 injections